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**Electronic and Ionic Transport in Polymers**

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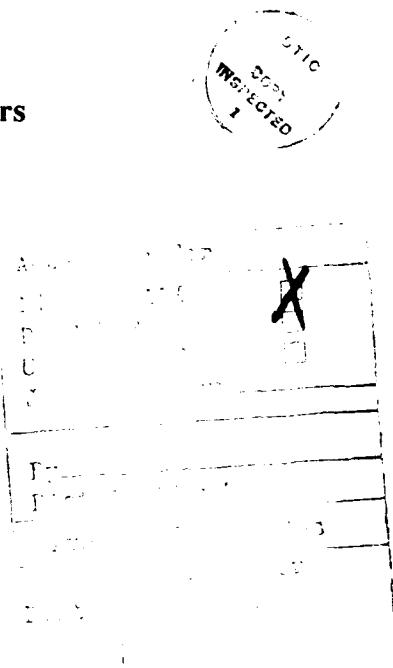
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## Electronic and Ionic Transport in Polymers

This research program has involved the study of processable, high molecular weight polyheterocycles, in particular polypyrroles and poly(3-alkylthiophenes) (where alkyl = butyl – dodecyl), both neat and as blends, as fibers, films and molded forms for their electronic, electromagnetic, ion transport and mechanical properties. Polypyrrole/polyelectrolyte molecular composites have been prepared electrochemically to gain insight into the effect of carrier polymer on the material's physical, mechanical, electrical and ion transport properties. New conjugated polymers, employing various aryl groups along the main chain and solubilizing substituents, were modeled theoretically, prepared synthetically, and structurally characterized to elucidate the effect of polymer structure on the optoelectronic properties. We have also studied electroactive polymers based on main chain transition metal complexes with the goal of controlling and improving physical and electronic properties of materials for specific applications. Fluoropolymers containing a high density of attached dipoles were examined with the goal of producing materials with elevated permittivities while retaining low loss behavior. A number of polymers containing phosphorus, nitrogen and organic groups in the main chain [poly(organo- $\lambda^5$ -phosphazenes)] have been prepared and studied. We have developed novel *in situ* techniques to monitor and quantify ion transport during redox switching of conducting polymers. Spectroscopy, electrochemistry and microgravimetry have been combined to develop a detailed understanding of the dynamics of the charge and ion transport through these conductive polymers. Theoretical calculations (PRDDO and ab initio) have been used to calculate structures, conformations, spin densities, HOMO-LUMO gaps, and potential energy surfaces for a wide variety of heterocyclic monomers and oligomers. They were used to predict the site of electrochemical polymerization and to understand how various substituents effect the electronic and three dimensional structure of the polymers. Extended Hückel band structure calculations were performed on many of these systems to assess qualitative trends in band gaps. Liquid crystalline polyethers and polyazomethines (LCP) were synthesized and characterized both for their mesomorphic and electroactive properties. Mesogenic core units which were incorporated in these LCP's included such potential electroactive centers as extended conjugated units and transition metal chelates.

The following is a brief summary of some of the results which we have achieved and studies which have been done over the past three years.

- 1) Preparation and study of self-doped conducting polymers that have ion-specific transport and are water soluble.

- 2) Use of a quartz crystal microbalance to analyze electropolymerization and charge transport in polyheterocycles.
- 3) Synthesis of variable conductivity copolymers of pyrrole and N-substituted pyrroles; substantiated with theoretical calculations.
- 4) The conductivity of polypyrrole free standing films and anion exchaged films have been shown to be relatively insensitive to dopant anion but dependent on morphology.
- 5) Spectroelectrochemical demonstration of proton transport through a Au-supported polypyrrole film during redox switching.
- 6) Theoretical calculations of pyrrole and thiophene monomers and oligomers correlate with electropolymerization tendencies.
- 7) Model compound studies have been carried out in the areas of high permittivity, ion-conducting and liquid crystalline polymers.
- 8) Charge transport properties of electrochemically prepared poly(3-methylthiophene) thin films were shown to depend on the morphology of the film.
- 9) Soluble transition metal bisdithiolene polymers were observed to be electroactive with three distinct oxidation states.
- 10) Carbon felt electrodes have allowed fairly large quantities of soluble poly(3-hexylthiophene) to be prepared.
- 11) Composite polypyrrole/poly(phenyleneterephthalamide propanesulfonate) films, where the dopant is a thermally stable polyelectrolyte modified Kevlar, were shown to have good electrical conductivities of ca.  $10^{-3}$  S cm $^{-1}$ .
- 12) The diffusion coefficients for anion exchange in polypyrrole were shown to be a function of polymer morphology.
- 13) Proton transport was seen to accompany switching of polypyrrole from the conducting to the insulating state.
- 14) Redox switching of polypyrrole has been observed to modify the luminescence behavior of an interfacial probe (e.g. pyrene).
- 15) Developed of quantum mechanical methods for estimating the relative energies of the quinoid and aromatic terms of polyheterocyclic systems.
- 16) The liquid crystalline properties of aliphatic/aromatic polyazomethine ethers was shown to depend on the configuration of atoms in the isomeric mesogenic core and the LC properties of some metal chelates depends on the nature of the transition metal.
- 17) A chemical polymerization method, using FeCl<sub>3</sub> and oxygen, has provided high molecular weight poly(3-alkylthiophenes), (alkyl = butyl – dodecyl) in fairly large quantities.
- 18) Fibers of poly(3-decylthiophene) have been melt spun in collaboration with Hoechst-Celanese, Summit, NJ, and completely characterized.

- 19) Molecular composite membranes of polypyrrole with poly(styrenesulfonate) have been prepared, shown to have good conductivity and mechanical properties and their ion transport properties have been studied using the electrochemical quartz crystal microbalance.
- 20) Large films of polypyrrole molecular composites have been incorporated into epoxy laminate panels and their surface resistivity monitored using microwave measurements in a collaborative program with General Dynamics, Fort Worth, TX.
- 21) We have demonstrated that the fluorescence intensity of a molecule located close to a conductive polymer (e.g. polypyrrole) surface may be modulated by redox switching, and accompanying variations in the dielectric characteristics of the polymer.
- 22) We have developed *in situ* optical absorption and fluorescent probes which can monitor ion transport during redox switching of polypyroles and polythiophenes.
- 23) We have used quantum mechanical calculations to do conformational analysis on intramolecularly hydrogen bonded copolymers containing heterocyclic and aromatic rings and to study the relative energies of aromatic and quinoid forms of polythiophene and related system. This has helped guide synthetic strategies.

The following is a listing of the journal publications and technical reports which have emanated from the contract.

#### Papers Published in Refereed Journals

- 1) Jang, G-W., Tsai, E. W. and Rajeshwar, K., "Electrochemically-Triggered pH Modulation at the Ruthenium Oxide/Electrolyte Interface: A Spectroelectrochemical Probe for the Proton Transport Mechanism", *J. Electrochem. Soc.*, **1987**, *134*, 2377.
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- 15) Ruiz, J. P., Reynolds, J. R., Nayak, K. and Marynick, D. S., "Soluble Ethylmercapto Substituted Polythiophenes", *Macromolecules*, **1989**, *22*, 1231.
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- 28) Shaffer, T. D. and Kramer, M. C., "Cyclization vs. Polymerization in Phase Transfer Catalyzed Polythioetherification", *Makromol. Chem.*, accepted for publication.
- 29) Nayak, K. and Marynick, D. S., "The Interplay Between the Geometric and Electronic Structures of Polyisothianaphthene and Polyisonaphthothiophene, Polythieno(3,4-b)pyrazine and Polythieno(3,4-b)quinoxaline", *Macromolecules*, accepted for publication.
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- 31) Sharma, S. C., Krishnamoorthy, S., Naidu, S. V., Eom, C. I., Krichene, S. and Reynolds, J. R., "Positron Annihilation and Conductivity Measurements on Poly(pyrole tosylate) and Poly(pyrrole fluoride)", *Phys. Rev. B.*, accepted for publication.

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- 32) Chien, J. C. W., Martinez, M. R., Reynolds, J. R. and Wnek, G. E., "Proton, Carbon-13, Nitrogen-15 NMR of Poly(pyrroles) and Poly(anilines)", *Macromolecules*, submitted for publication.
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- 35) Baker, C. K., Qiu, Y. J. and Reynolds, J. R., "Electrochemically Induced Mass Transport in Poly(pyrrole)/Poly(styrene sulfonate) Molecular Composites", *J. Am. Chem. Soc.*, submitted for publication.
- 36) Basak, S., Rajeshwar, K. and Kaneko, M., "Ion Binding of Poly{Pyrrole-Co[3-(Pyrrol-1-yl)Propane Sulfonate]} Thin Films", *Anal. Chem.*, submitted for publication.
- 37) Wang, F. and Reynolds, J. R., "Soluble and Electroactive Nickel Bis(dithiolene) Polymers Prepared Via Metal Complexation Polymerization", *Macromolecules*, submitted for publication.
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### Paper Acknowledging DARPA Support for Instrumental Purchase

- 39) Marques, H. M., Scooby, D. C., Victor, M. and Brown, K. L., "Optimisation of the Preparation and Purification of Three Monocarboxylic Acid Derivatives of Vitamin B<sub>12</sub> and Their Characterisation by <sup>13</sup>C NMR", *Inorg. Chim. Acta.*, **1989**, 162, 151.

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- 1) Pomerantz, M., Reynolds, J. R., Rajeshwar, K., Marynick, D. S. and Shaffer, T. D. "Electronic and Ionic Transport in Polymers", Quarterly-Technical Report No. 1, Grant No. N00014-86-K-0769 DARPA/ONR, December 12, 1986.
- 2) Pomerantz, M., Reynolds, J. R., Rajeshwar, K., Marynick, D. S. and Shaffer, T. D. "Electronic and Ionic Transport in Polymers", Quarterly-Technical Report No. 2, Grant No. N00014-86-K-0769 DARPA/ONR, March 13, 1987.
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- 4) Pomerantz, M., Reynolds, J. R., Rajeshwar, K, Marynick, D. S. and Shaffer, T. D. "Electronic and Ionic Transport in Polymers", End-of-the-Year Report No. 1, Grant No. N00014-86-K-0769 DARPA/ONR, July 22, 1987.
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- 21) Pomerantz, M., Reynolds, J. R., Rajeshwar, K, Marynick, D. S. and Shaffer, T. D. "Electronic and Ionic Transport in Polymers", Quarterly-Technical Report No. 16, Grant No. N00014-86-K-0769 DARPA/ONR, July 24, 1989.
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